



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/GB99/04010 <b>(22) International Filing Date:</b> 1 December 1999 (01.12.99) <b>(30) Priority Data:</b> 9826755.2                      4 December 1998 (04.12.98)                      GB <b>(71) Applicant (for all designated States except US):</b> BP CHEMICALS LIMITED [GB/GB]; Britannic House, 1 Finsbury Circus, London EC2M 7BA (GB). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> JONES, Michael, David [GB/GB]; 23 Westwood Road, Beverley, East Yorkshire HU17 8EN (GB). GRIMM, Seifried [DE/CH]; Neudorfstrasse 15, CH-8050 Zurich (CH). KEIM, Wilhelm [DE/DE]; Brusseler Ring 99, D-52074 Aachen (DE). WASSERSCHEID, Peter [DE/DE]; Grevenbroicher Strasse 2, D-50829 Koln (DE). <b>(74) Agent:</b> COLLINS, Frances, Mary; BP International Limited, Patents & Agreements, Chertsey Road, Sunbury on Thames, Middlesex TW16 7LN (GB).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> OLIGOMERISATION PROCESS  <b>(57) Abstract</b>  A process for the oligomerisation of C8-C36 alpha-olefins to give a polyolefin product comprising trimer which process comprises contacting the C8-C36 alpha-olefin with a catalyst in a solvent at a temperature below 20 °C wherein the catalyst comprises a complex of a chromium compound and a 1,3,5-triazacyclohexane and the polyolefin product comprises at least 70 mole% of trimer. The polyolefin product can be catalytically hydrogenated to lubricating oils.		

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### OLIGOMERISATION PROCESS

This invention relates to a process for oligomerising olefins and, in particular, to a process for the trimerisation of olefins using catalyst compositions comprising a source of chromium.

Methods of oligomerising unsaturated hydrocarbons including *inter alia*  
5 trimerising olefins using chromium containing catalysts are well known. For example, EP-A-0780353 describes a process for oligomerising olefins using a catalyst system comprising a chromium source, a pyrrole-containing compound and a metal alkyl. The catalyst system is supported on an inorganic oxide. Suitable trimerisation olefins are disclosed as C2-C30 olefins e.g ethylene, propylene, the butenes, 1-pentene, 4-methyl-1-  
10 pentene, 1-hexene, 1-heptene, the four normal octenes, the four normal nonenes, vinylcyclohexane and mixtures thereof. More specifically, this reference discloses the trimerisation of ethylene to 1-hexene, 1,3-butadiene to 1,5-cyclooctadiene, or the co-trimerisation of ethylene and hexene to form 1-decene or 1-tetradecene.

US 5137994 describes a method of producing a copolymer of ethylene and 1-  
15 hexene in which ethylene is simultaneously trimerised to 1-hexene which is copolymerised with further aliquots of ethylene in the presence of a bis-triarylsilyl chromate compound.

US 5087789 describes the oligomerisation of propylene and/or ethylene in the presence of a chromium catalyst on an aluminophosphate support and a selectivity  
20 modifier.

In an article entitled "Selective Trimerization of Olefins with 1,3,5-Triazacyclohexane Complexes of Chromium" by Randolph D Köhn et al based on a

presentation at "The XVIIIth International Conference on Organometallic Chemistry" held in Munich, Germany on 16-21 August 1998, the authors propose a mechanism for the trimerisation of 1-hexene using a 1,3,5-triazacyclohexane chromium catalyst activated with MAO.

5           Suprisingly, it has now been found that alpha-olefins having 8 to 36 carbon atoms can be oligomerised using a chromium based catalyst under specific reaction conditions to give a product predominating in trimers. The improved selectivity to trimers allows the product to be hydrogenated to lubricating oils.

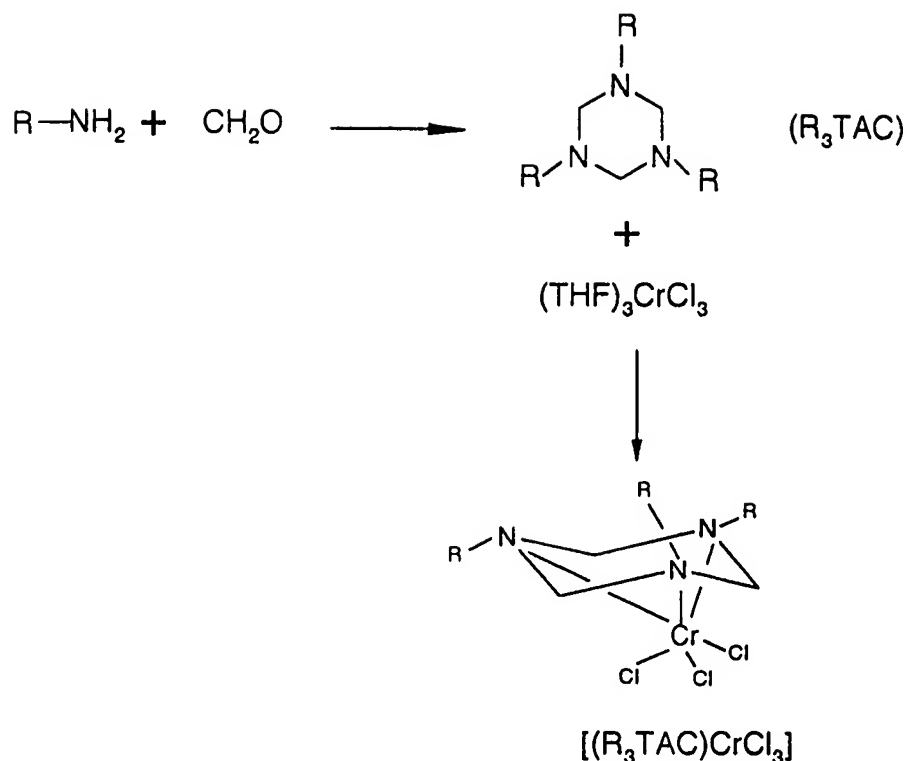
          Accordingly, the present invention provides a process for the oligomerisation of  
10 C8-C36 alpha-olefins to give a polyolefin product comprising trimer which process comprises contacting the C8-C36 alpha-olefin with a catalyst in a solvent at a temperature below 20°C wherein the catalyst comprises a complex of a chromium compound and a 1,3,5-triazacyclohexane and the polyolefin product comprises at least 70 mole% of trimer.

15           By trimer is meant a polyolefin product formed by the reaction of three alpha-olefin molecules. For example, 1-octene, 1-decene, 1-dodecene can trimerise to give olefin products containing 24, 30 and 36 carbon atoms respectively.

          The alpha-olefin that may be trimerised according to the process of the present invention preferably has 10 or more carbon atoms and more preferably has from 10-30  
20 carbon atoms. The alpha-olefin may be a straight or branched chain olefin. Specific examples of suitable alpha-olefins include octene-1, decene-1, dodecene-1, hexadecene-1, octadecene-1 and eicosene.

          The process of the present invention selectively converts an alpha-olefin to trimers. The selective conversion of alpha-olefin to trimer is preferably at least 80 mol%  
25 e.g 80-99 mol%, preferably at least 90 mol%, more preferably at least 95 mol%, especially at least 98 mol%. The yields of dimers, tetramers or other oligomers are reduced compared with known oligomerisation processes.

          The trimerisation catalyst used in the process of the present invention is a complex of a chromium compound and a 1,3,5-triazacyclohexane (hereinafter referred to  
30 as a chromium/triazacyclohexane catalyst). Preparation of such a complex is illustrated in the reaction scheme given below:-



The 1,3,5-triazacyclohexane may be prepared by known processes in the art e.g. by reacting a primary amine with formaldehyde. In the above reaction scheme the R group of the primary amine is suitably a hydrocarbyl group, such as an alkyl, cycloalkyl, aryl, alkaryl or aralkyl group. The hydrocarbyl group may have from 6 to 12 carbon atoms. Preferably, the hydrocarbyl group is selected from a dimethyl hexyl, n-octyl, methyl benzyl, dimethyl undecanyl and n-dodecanyl group. The R group in the 1,3,5-triazacyclohexane compound will correspond to the R group in the reactant amine. For example, where n-octyl amine is used as the amine reactant i.e. where R is n-octyl, the 1,3,5-triazacyclohexane compound produced will be tri-n-octyl-1,3,5-triazacyclohexane. This 1,3,5-triazacyclohexane compound will hereafter be abbreviated as "R<sub>3</sub>TAC" for convenience.

To produce a chromium complex of R<sub>3</sub>TAC, R<sub>3</sub>TAC is reacted with a chromium compound in the presence of a solvent. Suitable chromium compounds include those chromium compounds having labile ligands e.g. chromium halides such as chromium chlorides having labile ligands. Suitable labile ligands are those capable of being replaced by R<sub>3</sub>TAC e.g. tetrahydrofuran (THF). A suitable chromium compound is e.g. a complex of tetrahydrofuran with chromium trichloride ((THF)<sub>3</sub>CrCl<sub>3</sub>).

The solvent used for carrying out the reaction of  $R_3TAC$  with the chromium compound is suitably a saturated hydrocarbon e.g. an aliphatic or aromatic hydrocarbon. Preferably, the saturated hydrocarbon is n-hexane, cyclohexane, benzene or toluene. The reaction may be carried out by refluxing a solution containing the chromium compound and the  $R_3TAC$ . The resultant  $(R_3TAC)chromium$  complex can be recovered by removing the solvent under vacuum and purifying the product by column chromatography. In the above reaction scheme  $(THF)_3CrCl_3$  is refluxed with  $R_3TAC$  in e.g. equimolar amounts. The final chromium complex obtained and usable as a catalyst in the trimerisation process of the present invention is  $(R_3TAC)CrCl_3$ .

In the oligomerisation process of the present invention the alpha-olefin is contacted with a  $(R_3TAC)chromium$  complex in the presence of a solvent. The catalyst may be activated by a modifier such as an alkyl alumoxane. Preferably, the alumoxane is methyl alumoxane (MAO). The solvent is suitably a saturated hydrocarbon or an aromatic solvent which does not actively participate in the reaction. Examples of solvents that may be used include n-hexane, n-heptane, cyclohexane, benzene, toluene and the xylenes. The contacting of the alpha-olefin and the chromium/triazacyclohexane catalyst is suitably carried out in an atmosphere inert under the process conditions such as eg nitrogen, argon and the like.

The oligomerisation process is carried out at relatively low temperatures of less than  $20^{\circ}C$ , suitably in the range from about  $-30^{\circ}C$  to  $+20^{\circ}C$ , preferably in the range from about  $-25$  to  $+5^{\circ}C$ , eg  $0^{\circ}C$ . At temperatures of the order of about  $0^{\circ}C$ , the trimerisation reaction goes through to completion with minimum deactivation of the chromium/triazacyclohexane catalyst.

The process of the present invention may be carried out by initially mixing a solution of the alpha-olefin and the chromium/triazacyclohexane catalyst, cooling this solution down and then gradually adding a solution of the catalyst modifier to this mixture whilst allowing the reaction mixture to warm up. During the warming up of the reaction mixture, it may change colour. The reaction mixture so formed is then neutralised by the addition of a strong acid, eg hydrochloric acid thereto. This results in a biphasic mixture comprising an aqueous and an organic phase. The biphasic mixture is separated eg using a centrifuge to recover the organic phase. The organic phase is dried and the mol % of trimers in the polyolefin product is determined e.g. by gas

chromatography.

The polyolefin product may then be catalytically hydrogenated to form lubricating oils. The hydrogenation may be carried out in solution. The catalyst may be any suitable hydrogenation catalyst, but is preferably, a palladium catalyst supported on activated carbon or a Raney nickel catalyst. The hydrogenation is suitably carried out at elevated pressure, eg from about 2000-10000 KPa, preferably from about 4500-8000 KPa. The hydrogenation reaction is suitably carried out at a temperature in the range from 15-200°C, preferably from about 30-70°C. The duration of the hydrogenation reaction may be a few minutes to several days. After the hydrogenation reaction is complete, the reaction mixture is cooled down, depressurised and the solvent used removed by vacuum distillation. The purity of the hydrogenated product can be determined by gas chromatography and the viscosity of the resulting lubricant measured by rotary viscosimetry.

The nature of the hydrocarbyl group, R, in a catalyst complex such as  $(R_3TAC)CrCl_3$  does not seem to affect or influence the selectivity to the desired trimers but it does have some effect on the distribution of isomers in the trimeric product. This enables the viscosity of the lubricating oil to be controlled.

The process of the present invention is further illustrated with reference to the following Examples:

**A. Catalyst preparation**

Chromium compounds  $L_3CrCl_3$  (where  $L_3$  represents a tridentate ligand) are prepared by refluxing a solution of 0.1 mmol of a ligand  $L_3$  and 0.1 mmol  $(THF)_3CrCl_3$  (where THF represents a tetrahydrofuranyl group) in toluene (10 ml) for 1 hour. The solvent is thereafter removed under vacuum and the resultant chromium complex of THF is purified using column chromatography.

**B. Catalytic Experiments – General Procedure**

**General:** All experiments were carried out under argon as the inert gas atmosphere using standard Schlenk-technique.

**Catalysis experiments:** A chromium compound,  $L_3CrX_3$  (where X is a halogen, 0.05 mmol) were dissolved in toluene (10 ml). An internal standard (n-heptane, cyclohexane) was added where indicated to the resulting purple solution. 10 ml of the olefin to be trimerised were added and the reaction mixture was cooled down to -25°C using a

cooling bath. An MAO (methyl alumoxane) solution (10% in toluene, 5.0 mmol, ex Witco) was added dropwise to the above purple solution via a syringe. The reaction mixture was then allowed to warm up in a second bath to reaction temperature of 0°C so as to commence the trimerisation reaction and was stirred for the specified reaction time.

- 5 During the warming up, the mixture turned from purple to blue to green. The green solution was active in trimerisation of olefins. As the solution turns into brownish the activity decreases.

**Workup:** An aqueous solution of 2N HCl (10 ml) was added to the reaction mixture .

- The resulting biphasic mixture comprising an aqueous and an organic phase was  
10 separated using a centrifuge. The organic phase was recovered, dried and the conversion was determined using Gas Chromatography.

The results of these Examples are summarised in Tables 1.1 and 1.2 below in which the following abbreviations have been used:

- Me<sub>2</sub>Hex - Dimethyl hexyl  
15 Oc - n-Octyl  
MAO - Methyl alumoxane

### Catalytic experiments

**Table. 1.1: Reaction Conditions:**

Run No.	1	2
ligand	(Oc) <sub>3</sub> TAC	(Me <sub>2</sub> Hex) <sub>3</sub> TAC
n (L <sub>3</sub> CrCl <sub>3</sub> ) (mmol)	0.05	0.05
n MAO (mmol)	5.00	5.00
Standard (g)	n-heptane (0.45)	n-heptane (1.01)
Starting material (ml)	1-decene (10)	1-dodecene (20)
Solvent (ml)	toluene (10)	toluene (9)
Temp. (°C)	0	0



**Table.1.2 Results**

Run No.	<b>1</b>			
Time (hours)	3	23	48	
m (dimer)	0	0.01	0.03	
m (trimer)	0.02	2.4	7.71	
m (tetramer)	0	0	0	
mol% trimer *	100	98	92	

Run No.	<b>2</b>			
Time (hours)	0.4	2	4	16.5
m (dimer)	0	0	<0.01	<0.01
m (trimer)	0.19	0.57	5.17	7.00
m (tetramer)	0	0	0	0
mol% trimer *	99	99	95	93

5

\* based on the mol % of dimers and tetramers formed.

#### **Hydrogenation experiments:**

Trimers of 1-decene and 1-dodecene prepared by the process of the present invention using different chromium/triazacyclohexane catalysts were hydrogenated to lubricating oils. Any solvents and internal standards used were removed from the trimer product by vacuum distillation. The residual organic phase was diluted with the same amount of cyclohexane and a hydrogenation catalyst (Pd on activated carbon, 5 % Pd, ex Fluka, 0.01 g for 1 g of the trimerised product) was suspended in the solution of the organic phase in an autoclave. The autoclave was pressurised with hydrogen (7000 KPa (70 bar)) and heated up to 50°C for 18 hours. The reaction mixture was then cooled down to room temperature, the autoclave was depressurised, and the solvent was removed by vacuum distillation. The purity of the products was determined by Gas Chromatography and the viscosity thereof by rotary viscosimetry (ISO 3104: 1994). The viscosity, viscosity index (VI) and pour points of the lubricating oils are given in Table 2 below. In

the Table the C36 alkene product was obtained by the trimerisation of 1-dodecene and the C30 alkene products were obtained by the trimerisation of 1-decene. Table 2 also shows the viscosity, viscosity index (VI) and pour point measurements of polyalpha-olefin (PAO) lubricating oils. The PAO's are prepared by hydrogenation of the olefinic product resulting from the conventional oligomerisation of alpha-olefins using a BF<sub>3</sub>-alcohol catalyst. In Table 2, PAO(Feluy) is a commercially available polyalphaolefin from Feluy under the tradename Durasyn 164. From Table 2 it can be seen that the lubricating oils of the present invention have higher i.e. superior viscosity index (VI) than the polyalpha-olefins lubricating oils prepared by conventional processes. In the Table the following abbreviations were used:

Me<sub>2</sub>Hex                      1,5-dimethylhexyl  
 EtHex                        2-ethylhexyl  
 Bz                             Benzyl

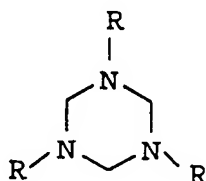
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Table 2

Lubricating Oil	Catalyst	Viscosity 40° C (cSt)	Viscosity 100° C (cSt)	VI	Pour Point micro
PAO	BF <sub>3</sub> -alcohol	17.5	4.0	124	-72
PAO (Feluy)	BF <sub>3</sub> -alcohol	17.0	3.9	120	-65
C36 alkene	(Me <sub>2</sub> Hex) <sub>3</sub> TACCl <sub>3</sub> /MAO	18.3	4.3	150	-30
C30 alkene	(Me <sub>2</sub> Hex) <sub>3</sub> TACCl <sub>3</sub> /MAO	12.2	3.2	126	-69
C30 alkene	(EtHex) <sub>3</sub> TACCl <sub>3</sub> /MAO	13.0	3.3	131	not measured
C30 alkene	(Me <sub>2</sub> Hex) <sub>3</sub> TACCl <sub>3</sub> /MAO	11.8	3.1	130	not measured
C30 alkene	(Bz) <sub>3</sub> TACCl <sub>3</sub> /MAO	12.3	3.2	126	not measured
C30 alkene	(Bz) <sub>3</sub> TACCl <sub>3</sub> /co-MAO	12.1	3.2	137	-60

## Claims:

1. A process for the oligomerisation of C8-C36 alpha-olefins to give a polyolefin product comprising trimer which process comprises contacting the C8-C36 alpha-olefin with a catalyst in a solvent at a temperature below 20°C wherein the catalyst comprises a complex of a chromium compound and a 1,3,5-triazacyclohexane and the polyolefin product comprises at least 70 mole% of trimer.
2. A process according to claim 1 wherein the C8-C36 alpha-olefin is selected from the group consisting of octene-1, decene-1, dodecene-1, hexadecene-1, octadecene-1 and eicosene
3. A process according to claim 1 or claim 2 wherein the polyolefin product comprises at least 80% of trimer.
4. A process according to claim 3 wherein the polyolefin product comprises at least 90% of trimer.
5. A process according to any one of claims 1 to 4 wherein the 1,3,5-triazacyclohexane is of the formula



wherein R is a hydrocarbyl group.

6. A process according to claim 5 wherein R is selected from the group consisting

of dimethylhexyl, n-octyl, methylbenzyl, dimethylundecanyl and n-dodecanyl

7. A process according to any one of claims 1 to 6 wherein the catalyst is a complex of a 1,3,5-triazacyclohexane and a chromium halide

8. A process according to claim 7 wherein the chromium halide is a chromium  
5 chloride.

9. A process according to any one of claims 1 to 8 wherein the catalyst additionally comprises an alkyl alumoxane.

10. A process according to any one of claims 1 to 9 wherein the process is carried out at a temperature of from -25 to 5°C

10 11. A polyolefin product produced by the process of any one of claims 1 to 10

12. A process for producing a lubricating oil wherein said process comprises catalytically hydrogenating a polyolefin product produced by any one of claims 1 to 10

13. Use of a polyolefin product produced according to any one of claims 1 to 10 in the manufacture of a lubricating oil.

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**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 C07C2/32 C10G50/02

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
 IPC 7 C07C C10G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 744 677 A (WU FENG-JUNG) 28 April 1998 (1998-04-28) claims	1
A	EP 0 583 072 A (BP CHEMICALS) 16 February 1994 (1994-02-16) claims	12, 13

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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